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Adsorption of clofibric acid by the activated carbon prepared with polyester cloth waste: Study of the operational parameters, kinetic and adsorptive equilibrium using the non-linear method

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Abstract: The objective of this research work is to examine the feasibility of preparing adsorbent materials from textile waste (polyester) for the elimination of pharmaceutical products such as clofibric acid (CA). The results showed that the adsorbents prepared by chemical activation in the presence of phosphoric acid followed by pyrolysis at 600 °C conduct to microporous materials with large specific surfaces. Batch experiments were conducted to study the effect of contact time, initial CA concentration, solution pH and temperature. Elimination yields by adsorption of CA in aqueous solution greater than 95% are obtained with dilute solutions (10 mg L^{-1}) at room temperature and at pH=3. The adsorption kinetic is perfectly described by the pseudo-second-order model and the isotherms are of Freundlich types. The results indicate that this process is spontaneous, efficient and potentially applicable in the removal of CA from water.

Keywords: activated carbon cloth; pharmaceutical active compounds; waste water; H_3PO_4.

INTRODUCTION

Pharmaceutical active compounds (PhACs) are a class of emergent pollutants which are being continuously introduced in the environment mainly due to improper disposal of unused or expired drugs or through excretion and inefficient removal in sewage treatment plants (STPs).[1] Even though the amounts found in the environment are usually low (they are often detected in trace concentrations (ng L^{-1}), long-term exposure of aquatic and terrestrial organisms may provoke adverse effects in respective ecosystems.[2] Some of these compounds such as carbamazepine, clofibric acid, diclofenac, tetracycline, paracetamol and caffeine

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have shown high persistency behavior which may lead to its bioaccumulation when present in the environment. Moreover, the degradation of some drugs can produce highly toxic and carcinogenic compounds.

It is thus important to remove all these pharmaceutical products from wastewater and this can be done using electrochemical method, membrane filtration, adsorption, biodegradation and advanced oxidation processes (AOPS). Compared with the above methods, adsorption is considered as a promising method for removing various pollutants from wastewater due to its economical, renewable, and flexible operation.[3]

Activated carbon cloths (ACCs) present technological advantages over more traditional powder or granular forms of activated carbons, including high adsorption capacity, uniform porosity, high rates of adsorption/desorption from the gas or liquid phase as well as of new applications, as molecular sieves, catalysts or electrodes.[3,4] The textual and chemical characteristics of the activated carbon depend on the nature of the precursor used as well as the methods and conditions of production.[5] Currently, the major precursors for producing ACC are composed of synthetic materials such as acrylic, nylon and polyester fibres and natural materials such as wool, flax, viscose and cotton.[3] The preparation of activated carbon from polyester woven waste, similar to that of conventional activated carbon, involves its treatment by physical or chemical activation processes.[6]

Some researchers have shown their efficiency in aqueous medium for the removal of organic and inorganic compounds for example Brasquet et al.[7] examined the quantitative structure–property relationship for the adsorption of 55 organic compounds onto activated carbon cloth. Brasquet and Cloirec.[8] studied the effects of activated carbon cloth surface on organic adsorption in aqueous solutions. Ayranci and Dumain.[9] evaluated the adsorption capacity of ACCs vis-avis of phenolic compounds (phenol, hydroquinone, *m*-cresol, *p*-cresol and *p*-nitrophenol). Adsorption of cadmium by activated carbon cloth based on polyacrylonitrile fibre as a precursor was oxidised using nitric acid, ozone and electrochemical oxidation has been reported by Rangel-Mendez and Streat.[10] . Álvarez-Merino et al.[11] investigated the adsorption of Zn (II) ions from aqueous solution under static conditions was studied using commercial activated carbons in the form of grains and cloth. Akkouche et al.[12] Studied of the adsorption of tetracycline and paracetamol from aqueous solutions using activated carbon derived from cotton textile waste modified with H_3PO_4. Boudrahem et al.[3] studied the adsorption of clofibric acid from aqueous solutions using activated carbon derived from cotton textile waste activated with H_3PO_4.

In this work we investigated the feasibility of polyester cloth waste to be used as precursor for production of activated carbons with activation by H_3PO_4. The physicochemical properties of ACs, such as BET surface area, morphology and surface functional groups, were analyzed to better understand the mechanism of adsorption of clofibric acid (2-(p-chlorophenoxy)-2-methylpropionic acid, CA). The effects of initial concentration of adsorbate, pH of the solutions, contact time and temperature were evaluated. Moreover, different adsorption isotherm models including Langmuir and Freundlich were used to analyze the adsorption equilibrium data. Pseudo-first order, pseudo-second order models were used to study the kinetic process.

The choice of textile waste in this study is linked to its great availability, its low cost and the need to preserve natural resources for future generations. For example, it is estimated that the production of textile waste in Algeria for the year 2014 at 1,430,000 tons. As for the choice of the target molecule (clofibric acid), this is due to its belonging to a class of consumer products, to its presence in the environment and its specific action on microorganisms.

EXPERIMENTAL

Materials

Polyester cloth waste used was provided from the clothing production factory (ALCOST-Bejaia-Algeria). Nitrogen gas was industrial grade of 99% purity. The reagent grade chemicals used in the study (H_3PO_4, H_2SO_4, HCl, NaOH) and clofibric acid (CA), were purchased from Aldrich and Junsei chemical companies.

Preparation of the activated carbon (AC)

Polyester cloths residue were used as a precursor for preparation of the activated carbon (AC). The cloths previously weighed were immersed in the H_3PO_4 solution during 7 h at 85 °C to ensure the access of activating agent to the interior of the precursor, and then the temperature of the mixture was increased and maintained to 100°C until it is completely dry. Three activation ratios (25, 50 and 75% wt) (wt = mass of H_3PO_4/mass of precursor) were tested. The samples dried were pyrolysed at 600°C during 1h under an inert gas stream (N_2 flow). The heating rate was 10 °C min^{-1}. The ACs samples were cooled down to room temperature still under N_2 flow. Afterwards, the resulting activated carbons were rinsed thoroughly with distilled hot water until neutral pH was obtained in order to remove all acid, then dried and kept in a desiccator before use.

Characterization of the activated carbon prepared

The surface area, micropore volume and pore size distribution were determined using the nitrogen adsorption isotherm technique measurements at the liquid nitrogen temperature of 77 K (Micromeritics Instrument Corporation MicroActive Tristar II 3020).

The surface morphology of ACs was examined by scanning electron microscopy (SEMJSM820, Jeol Ltd., Japan).

Surface chemical properties of the samples were characterized by the Fourier Transform Infrared spectroscopy (Shimadzu FTIR-8300, Japan) at room temperature. The spectra were performed in the 4000– 400 cm^{-1} wave number range.

The carbon surface charge is mainly determined by the pH of the adsorbate solution. The pH of the point of zero charge (pH_{PZC}) of the carbon was determined using the method reported by Khenniche and Aissani.[13]

Batch adsorption procedure

Adsorption experiments were carried out by mixing 0.25 g of ACs with 250 mL of each clofibric acid solution (10 to 100) mg L^{-1} in a batch reactor (500 mL) under the following conditions: 360 rpm stirring speed, 3 h contact time, temperature (20, 30, 40 and 50) °C and desired initial pH value of the solution. The concentration of the clofibric acid was measured using UV-Vis spectrophotometer at 227 nm wavelength. The CA adsorption capacity at time *t*, q_t, was evaluated using the equation (1):

$$q_t = \frac{(C_0 - C_t)V}{m} \tag{1}$$

in which q_t is the adsorption capacity at time t (mg g^{-1}), C_0 (mg L^{-1}) is the initial concentration of CA, C_t (mg L^{-1}) is the concentration of CA at time t, V (L) is the volume of the CA solution and m (g) is the mass of the ACs.

At the equilibrium: $q_t = q_e$ and $C_t = C_e$

RESULTS AND DISCUSSION

Adsorbent characteristics

Nitrogen adsorption- textural characteristics

Before attempting to obtain quantitative information, a study of form and textural characteristics is imposed. Nitrogen adsorption-desorption isotherms are plotted for the various adsorbents prepared by plotting the amount of nitrogen adsorbed or desorbed per gram of adsorbent as a function of relative pressure. The results presented in the Fig. 1 show that all nitrogen adsorption isotherms are type I (Langmuir isotherm) according to the IUPAC classification.[14] This type of isotherm suggests that the adsorbents are of the microporous type, this result is confirmed by the desorption isotherm. The speed of reaching the plateau is an indication of the pore size distribution and the presence of the horizontal plateau suggests a very low external surface area.

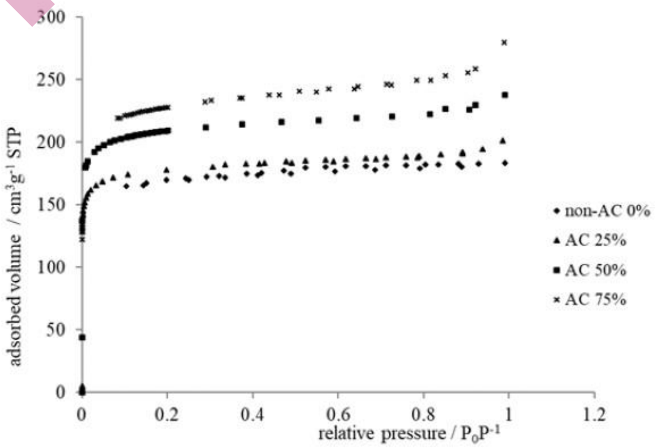


Fig. 1. Adsorption-desorption isotherms of nitrogen at 77 K of ACs with different H_3PO_4 impregnation ratio.

The physical properties results from the N_2 adsorption-desorption isotherms of all ACs were determined and reported in Table I

Analysis of the Table I data reveals that activated carbons prepared from polyester have characterized by large specific surfaces. The activation increases the S_{BET}. V_t, V_{mic} and V_{mes}. The microporosity ratio (V_{mic}/V_t) % of the adsorbents prepared decreases with impregnation ratio since it goes from 99.74 % for non-activated carbon 0% H_3PO_4 to 98.6 % for an activation rate of 75% H_3PO_4. We notice in this case a development of mesoporosity. This effect may be due to the hydrolysis of the polyester during the impregnation with the acid, which means a important release of volatile compounds during the heat treatment.[15] These results are confirmed by Ramos *et al.,*[15] showing that the use of phosphoric acid as an activating agent not only contributes to the creation of new micropores but also to the enlargement of the pores already existing in the precursor.

TABLE I. Physico-chemical properties of activated carbons

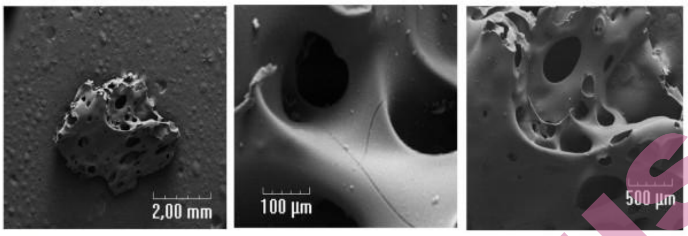
	Impregation ratio with H_3PO_4			
Parameters	AC 0%	AC 25%	AC 50%	AC 75%
V_{tot} / cm^3g^{-1}	0.271	0.335	0.285	0.369
V_{mic} / cm^3g^{-1}	0.271	0.326	0.274	0.363
Microporosity /V_{mic}/V_{tot}/%	99.74	97.41	96.18	98.6
V_{mes} / cm^3g^{-1}	0.00072	0.0087	0.011	0.0052
Mesoporosity / V_{mes}/V_{tot}/%	0.264	2.591	3.819	1.4
S_{BET} / m^2g^{-1}	415.45	776.23	534.08	826.2
S_{ext} / m^2g^{-}	89,17	101.21	75.92	152.2
S_{mic} / m^2 g^{-1}	326.28	675.02	458.16	674
$d_p = 4V_{tot}/S$ / Å	26.13	17.25	21.32	17.85
pH_{pzc}	5.1	4.25	4	3.9
$q_{e.}$ / mg g^{-1}	34.45	63.66	75.39	80.46

Morphological characterization of the ACs

The scanning electron micrographs (SEM) of the non-activated carbon (non-AC 0%) and the AC 75% are presented in Fig. 2 (a-b). The non-activated carbon (0% H_3PO_4) and activated carbon (75% H_3PO_4) samples derived from the polyester precursor show visible signs of fibers collapse and breakage, likely due to an intensified reaction with the polyester caused by the acid in the impregnation stage and the pyrolysed temperature, with the woven form of the precursor is gone, unlike carbon prepared from cotton, it has kept its woven and fibrous character.[3]

It is noticed also that the porous structure (size of the pores) is well-developed, containing different sizes and shapes of pores which result from the activation process.

a) Non AC 0%



b) AC 75%

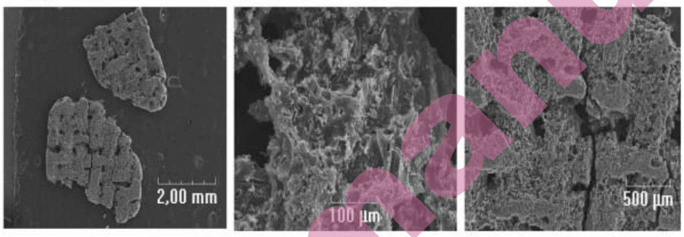


Fig. 2. SEM micrograph: a) of The non-activated carbon (0% H_3PO_4) and b) of activated carbon (75% H_3PO_4).

FTIR analysis

The FTIR spectra of the ACs prepared with different ratio (0, 25, 50 and 75) % are presented in Fig. 3. It is noted that the spectra corresponding to the different adsorbents are similar with respect to the type of functions. The difference is only in the intensity of the peaks. The higher the activation rate, the more intense the peaks. Examination of all these spectra reveals the following absorption bands:

A broad absorption band observed between 3600–3300 cm^{-1} with a maximum around 3400 cm^{-1} is characteristic of the hydrogen elongation vibration of hydroxyl groups (of carboxyls, phenols or alcohols) and water adsorbed by the materials analyzed.[16]

The peaks at approximately 2300, 2370 cm^{-1} is characteristic of the C≡C stretching vibration of alkyne groups.[17]

The new band appearing in the AC 25%, AC 50% and AC 75% around 1715 $cm^{-1}$and 1600 cm^{-1} observed on the spectrum of non-AC 0%, is most likely due to the C=O indicating the formation of carbonyl-containing groups (ketones, aldehydes, lactones, and carboxyl groups).[18]

The peak at 1556 cm^{-1} is a characteristic of the C=O stretch of the carbonyl group in a quinone and represents the γ-pyrone structure with strong vibrations from a combination of C=O and C-C.[19]

The broad band at 1300–500 cm^{-1} was assigned to the C–O stretching and O–H bending modes such as phenols, alcohols, esters and carboxylic acids.[20]

The presence of hydroxyl groups of phenolic and carboxylic gives an acidic character to activated carbon surface whereas carbonyl and quinone groups confer a basic character to the adsorbent surface.

The pH PZC (Table I) is shifted towards lower pH values when the impregnation ratio increases due to the introduction of acidic groups.

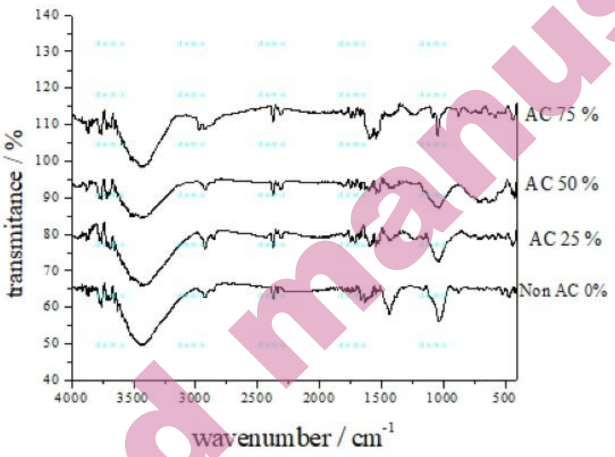


Fig. 3. FTIR spectra of the carbon.

Effect of impregnation ratio on adsorption amount of clofibric acid

The impregnation ratio variation effect on the adsorption kinetics of the CA onto activated carbon is presented in Table I. A higher elimination of CA is observed when the impregnation ratio is increased from 0% to 75% of H_3PO_4. The best adsorption rate (% removal) of activated carbon obtained at 75% phosphoric acid is 80.46 mg g^{-1}. This may be attributed to the increase in adsorbent surface area, microporosity development (≈ 99%) and availability of more adsorption sites resulting from the increase in impregnation ratio. Consequently, the activated carbon prepared with an activation ratio of 75% H_3PO_4 was used in all subsequent experiments.

Effect of pH on the removal of clofibric acid

pH is an important factor in any adsorption study. It can condition both the surface charge of the adsorbent as well as the structure of the adsorbate. This quantity characterizes the water, and its value will depend on the origin of the effluent. The treatment technique to be adopted will strongly depend on the pH value. This is the reason why, in any study of adsorption, the study of the influence of pH on the adsorption capacity of a given solute on a specific adsorbent is essential.

The kinetic results of the adsorption of clofibric acid showed that the pH studied in the range 3-9 is a critical factor. From Fig. 4a, it appears that CA elimination is best at a very acidic pH (pH=3). The adsorption capacity gradually decreases when the pH increases. It reaches its minimum at basic pH (pH = 9). A similar value was found in a previous study, adsorption of clofibric acid by an activated carbon prepared from cotton [3].

The pH_{PZC} of AC 75% is 3.9, then the surface carries a positive charge at pH solution less than 3.9, neutral for pH = pH_{PZC} and negative for solution pH above 3.9.

The increase of the CA adsorption capacity with increasing the acidic degree of the solution is attributed to the anionic and molecular forms of the CA and the positive surface charge of the activated carbon at lower pH. Therefore, the anionic form of CA is attracted by the positive charges of the activated carbon surface area. When the pH increases ($pH > pH_{PZC}$), the surface would be more negatively charged and above the pKa value clofibric acid is in anionic form which results in high electrostatic repulsions, leading to no significant adsorption.

Effect of contact time and initial clofibric acid concentration

One of the factors known his influence the amount of solute removed by adsorption is the adsorbent-adsorbate contact time. We therefore monitored the CA adsorption capacity over a period of 3 hours for different initial acid concentrations.

Fig. 4b indicates that equilibrium is reached almost after 60 minutes. It also shows that the adsorption takes place in two stages. At the start of the experiment, adsorption is rapid, which is due to the high availability of vacant active sites on the surface of the adsorbent. This step is followed by a second, slower step as there are fewer and fewer active sites to which clofibric acid binds. The acid adsorption capacity tends to stabilize, which is evidenced by the appearance of a plateau. Given these results, we set the duration of our experiences at 180 min to make sure there is no desorption of adsorbed molecules in case of important contact times.

The influence of initial CA concentration on the adsorption capacity and kinetics is shown in Figure 4b. The results show that the CA adsorption capacity increases with increasing initial solution concentration. This development can be explained by the existence of a strong gradient in the concentration of CA which increases between the solution and the surface of the adsorbent when C_0 increases.

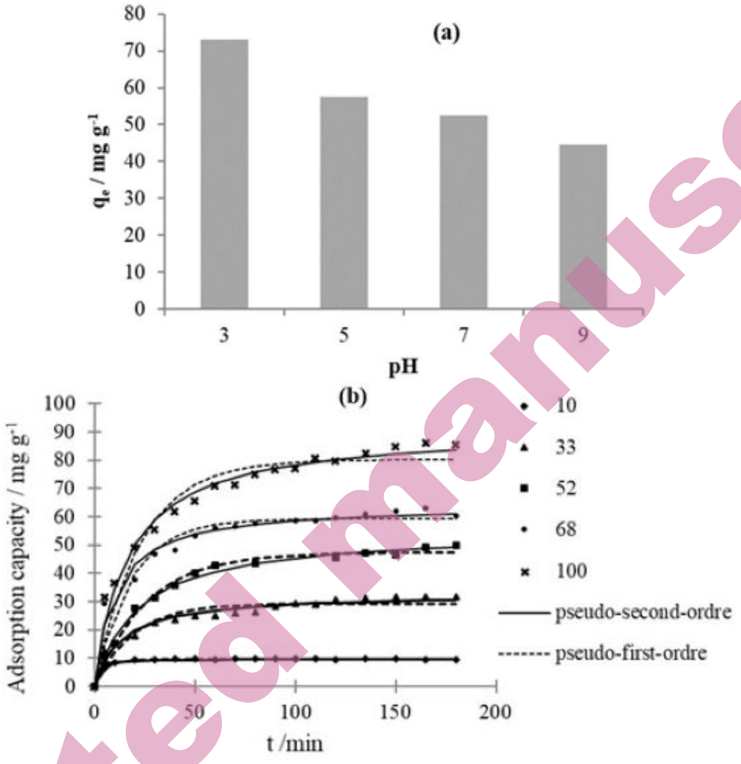


Fig. 4. Adsorption of CA onto AC 75% at Different: (a) initial pH; Conditions: AC 75%, agitation speed= 360 rpm and T= 20 °C, (b) contact time and initial CA concentration; Conditions: AC 75%, agitation speed= 360 rpm; T=20°C and pH=3.

Adsorption kinetic

In order to examine the mechanism of the adsorption process, several models are given in the literature. We have tested three kinetic models in particular to analyze our experimental results: the pseudo-first-order model (equation 2), the pseudo-second-order model (equation 3) and the intra-particle diffusion model (equation 4).[3]

$$q_t = q_e\left(1 - e^{-k_1 t}\right) \tag{2}$$

$$q_t = \frac{q_e^2 k_2 t}{1 + q_e k_2 t} \tag{3}$$

$$q_t = x_i + k_d t^{0.5} \tag{4}$$

Where q_e and q_t (mg g^{-1}) are the adsorption capacity at equilibrium and at time t, respectively, k_1 (min^{-1}), k_2 (g mg^{-1} min^{-1}) and k_d (g mg^{-1} min^{-1}) are the rate constant of the pseudo-first-order, pseudo-second-order and intra-particular diffusion equations, respectively and x_i is the intercept of the straight line which is related to the boundary-layer thickness.

All the constants of the models tested (Table II) were determined by maximizing the error function and using the solver add-in with Microsoft's spreadsheet, Microsoft Excel.[21] The error function (coefficient of determination, r^2) (Eq. (5)) employed were as follow:

$$R^2 = \frac{\sum(q_{cal}-\bar{q}_t)^2}{\sum(q_{cal}-\bar{q}_t)^2+\sum(q_{cal}-q_t)^2} \tag{5}$$

Where q_{cal} and q_t both expressed in mg g^{-1}, represent the adsorption capacities of clofibric acid (CA) onto AC 75% at time t. q_{cal} is obtained from the model, while q_t is determined experimentally. $\bar{q}_t$ denotes the average of the experimental values (q_t).

According to Fig. 4b and the values of the parameters presented in Table II the high R^2 and the difference between $q_{e(cal)}$ and $q_{e(exp)}$ values, the adsorption kinetics of CA onto AC 75% is described by a pseudo-second-order model. The matching adsorption process to the pseudo-second-order model indicated that various mechanisms such as chemisorption and diffusion into the pores were contributed in the adsorption of CA onto active sites of the adsorbent.[22]

Fig. S-1 in the Supplementary material shows two distinct linear segments, indicating a two-step adsorption process. The first linear portion corresponds to adsorption on the external surface (film diffusion), which is considered a fast step. The second portion is attributed to intra-particle diffusion, representing a slower phase of the adsorption process. The analysis of the intra-particle diffusion model (Table II) demonstrated that this diffusion is not the rate-limiting mechanism, and that diffusion through the boundary layer surrounding the adsorbent plays a non-negligible role.

Adsorption isotherms

Isotherms comprise an essential part of adsorption studies. From them, it is possible to evaluate the physical interactions between adsorbate and adsorbent.[23]

In the present study, the Langmuir and Freundlich models (Table III) were tested by using non-linear method to evaluate the adsorption capacity of our adsorbent and to determine the equilibrium isotherm. Fig. 5 shows the experimental data for the adsorption of CA on AC 75% at different temperature and the predicted equilibrium curves.

The adsorption isotherms obtained (Fig. 5) have a similar appearance and correspond to type L, according to the classification of Giles et al.[24]. This type of isotherm suggests that the molecules adsorb flat on the surface of the adsorbent, and that there is no competition between clofibric acid and water molecules for the adsorption sites.

It can be deduced (Fig. 5 and table III) that the equilibrium data are well described by the Freundlich isotherm model.[25] The fitting results show that the value of *n* is superior to 2 at the temperature (293 and 303 K), indicating that the adsorption is good, while at the temperature of 313 K (n=0.9) and 323 (n=1.36) are poor and moderately difficult respectively.

TABLE II. Kinetic parameters for adsorption of CA onto AC 75%

				Pseudo-second-order kinetics		
C_0 / mg L^{-1}	$q_{e\,exp}$ / mg g^{-1}	$q_{e\,cal}$ / mg g^{-1}	k_1 / L min^{-1}	$q_{e\,cal}$ / mg g^{-1}	k_2 / g mg^{-1} min^{-1}	R^2

		C_0 / mg L^{-1}				
					0.048	

k_{d1} and k_{d2} in g mg^{-1} min^{-1}

A comparison is made between the adsorption capacity of AC 75% for removal of pharmaceutical compound (CA) and other adsorbents reported in the literature for example:

Mestre *et al.*, 2010[26] studied the adsorption of clofibric acid (pKa 3.6) from aqueous solutions using two activated carbons derived from cork waste: CAC (chemically activated with K_2CO_3) and CPAC (physically activated from CAC via steam treatment). Textural analyses showed that CAC is predominantly microporous, with 68% of its microporous volume consisting of narrow micropores, while CPAC exhibited both micropores and more developed mesoporosity. CPAC had the highest specific surface area (1060 m^2 g^{-1}) and total porosity. Kinetic data were modeled using a pseudo-second-order kinetic model. The study demonstrated that pH significantly influenced clofibric acid adsorption, with peak efficiency at pH 2, followed by progressive declines at pH 3.6 and pH 5. The sigmoidal adsorption isotherms were fitted with the Dubinin–Astakhov model, revealing a maximum adsorption capacity of 295 mg g^{-1} for CPAC.

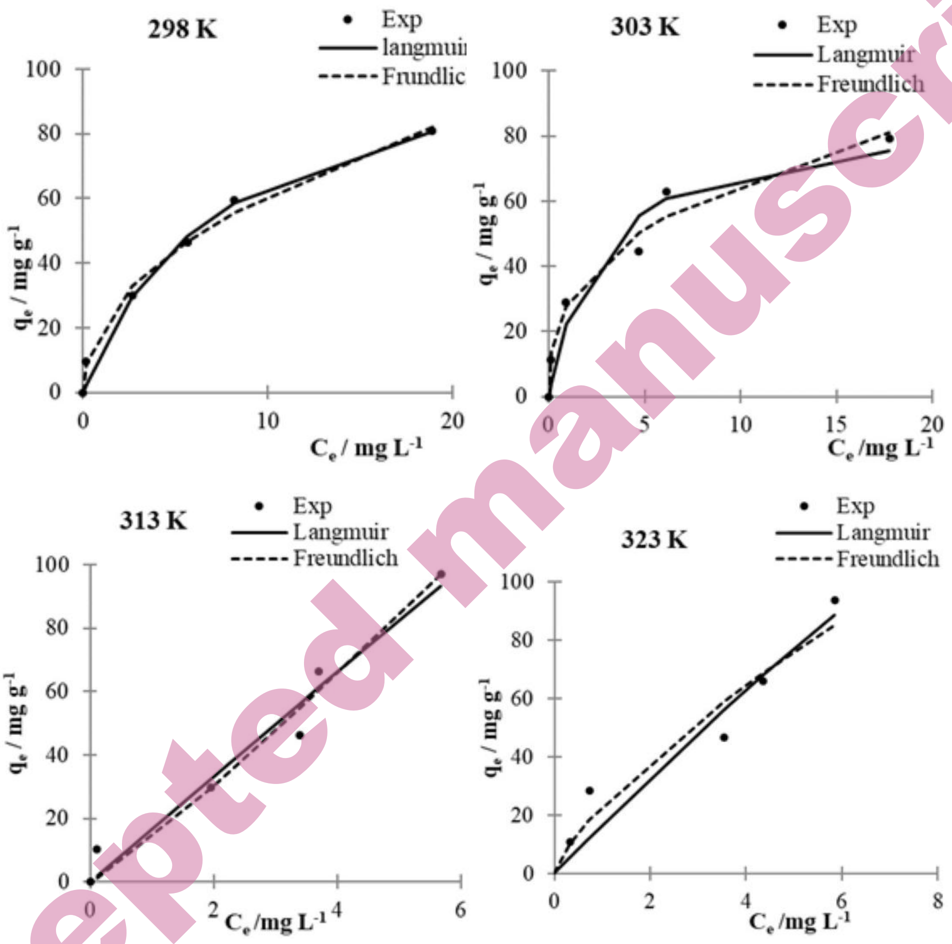


Fig. 5. Langmuir and Freundlich isotherm for the adsorption of CA onto AC75% (conditions: 0.25 adsorbent, 250 mL of adsorbate solution, adsorption time 180 min, agitation speed 360 rpm, pH 3 and temperature 293-323 K).

TABLE III. Parameters of the Langmuir and Freundlich models for the Adsorption of CA onto ACC 75 %

model	Ref.	Parameter	293	303 K	313 K	323 K
Langmuir		q_m / mg g^{-1}	112.71	86.94	3868	803.66
$q_e = \dfrac{q_m K_L C_e}{1 + K_L C_e}$	19	K_L / L mg^{-1}	0.132	0.38	0.004	0.02
		R^2	0.98	0.95	0.96	0.94
		K_f / mg$^{-1/n}$ L$^{1/n}$	20.75	28.94	13.95	23.25
Freundlich		1/n	0.47	0.36	1.11	0.73
$q_e = K_F C_e^{1/n}$	19	N	2.22	2.77	0.9	1.36
		R^2	0.99	0.97	0.97	0.95

q_m = maximum adsorption capacity; K_L = Langmuir constant; K_F and n = Freundlich constants

Roza *et al.* (2014)[27] investigated the comparative adsorption behavior of ibuprofen (IBP) and clofibric acid (CA) onto activated carbon derived from bamboo waste, prepared by chemical activation with $ZnCl_2$ followed by microwave heating (ABW). Textural analyses revealed that ABW exhibits a porous structure combining micropores and mesopores (type II isotherm with a low-pressure hysteresis loop), and a specific surface area of 722.27 m^2g^{-1}. The adsorption of both compounds was more effective under acidic conditions, particularly at pH values between 2 and 5, due to favorable electrostatic interactions. The adsorption kinetics followed a pseudo-second-order model, suggesting that the process is governed by both chemical adsorption and intraparticle diffusion. The adsorption isotherms were best described by the Langmuir model, indicating monolayer adsorption, with maximum adsorption capacities of 278.55 mg g^{-1} f or IBP and 229.35 mg g^{-1} for CA. The calculated Gibbs free energy values (-6.15 kJ mol^{-1} for IBP and -5.56 kJ mol^{-1} for CA) indicate that the adsorption processes are spontaneous and thermodynamically favorable.

Lu *et al.* (2016)[28] studied the adsorption and removal of clofibric acid (CA) and diclofenac (DCF) from water using a magnetic ion exchange (MIEX) resin. Adsorption was found to be optimal within a pH range of 5 to 9, where both compounds predominantly exist in their anionic forms, thus favoring their exchange with the quaternary ammonium groups on the resin. The kinetic data fit primarily the pseudo-first-order model, and the study revealed that the process is jointly controlled by external mass transfer and surface diffusion. The maximum adsorption capacities reported were 133.69 mg g^{-1} for CA and 322.31 mg g^{-1} for DCF.

Hasan *et al.* (2012)[29] investigated the removal of clofibric acid (CA) from water by adsorption using metal-organic frameworks (MOFs), particularly MIL-101, which they compared to activated carbon. MIL-101 exhibited a higher maximum adsorption capacity (312 mg g^{-1} versus 244 mg g^{-1} for activated carbon), which is attributed to its high specific surface area (~3100 m^2g^{-1}) and well-developed porosity. The adsorption process follows a pseudo-second-order kinetic model, with equilibrium reached more rapidly using MIL-101 than with the other adsorbents. Furthermore, the adsorption is strongly pH-dependent: it is more effective under acidic conditions, suggesting a favorable electrostatic interaction between the anionic functional groups of CA and the cationic sites of the MOF.

A detailed comparison of all the discussed adsorbents is presented in Table S-I of Supplementary material.

CONCLUSION

This study demonstrated that polyester textile waste can be effectively valorized into high-performance adsorbent materials for the removal of a pharmaceutical pollutant, clofibric acid, present in wastewater. The adsorbent obtained through chemical activation with phosphoric acid (H_3PO_4) followed by pyrolysis at 600 °C resulted in a microporous activated carbon with a high specific surface area (up to 826 m^2 g^{-1} for a 75% impregnation ratio). Textural analyses (Type I isotherms) and morphological characterizations (SEM) confirmed the dominance of microporosity, while FTIR spectroscopy revealed the presence of functional groups (C=O, -OH) that enhance adsorption.

Adsorption tests showed:

- a removal efficiency greater than 95% for low CA concentrations (10 mg L^{-1}),
- adsorption kinetics described by the pseudo-second-order model, indicating chemisorption-type interactions,
- optimal adsorption at pH 3, highlighting the importance of the carbon surface charge and the ionic form of the pollutant,
- equilibrium data well-fitted by the Freundlich isotherm model, suggesting heterogeneous adsorption on various types of sites,
- the most efficient sample corresponded to an impregnation ratio of 75% H_3PO_4, for which the maximum measured adsorption capacity reached 80.46 mg g^{-1}.

From an environmental perspective, this research offers an economical and sustainable solution for managing textile waste and treating water contaminated with pharmaceutical compounds. The produced ACs show promising potential for large-scale applications, particularly in hospital or industrial wastewater treatment.

SUPPLEMENTARY MATERIAL

Additional data are available electronically at the pages of journal website: https://www.shd-pub.org.rs/index.php/JSCS/article/view/13381, or from the corresponding author on request.

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АДСОРПЦИЈА КЛОФИБРИНСКЕ КИСЕЛИНЕ АКТИВНИМ УГЉЕМ ДОБИЈЕНИМ ОД ОТПАДНЕ ПОЛИЕСТЕРСКЕ ТКАНИНЕ: ИСПИТИВАЊЕ ЕКСПЕРИМЕНТАЛНИХ ПАРАМЕТАРА, КИНЕТИЧКЕ И АДСОРПЦИОНЕ РАВНОТЕЖЕ ПРИМЕНОМ НЕЛИНЕАРНЕ МЕТОДЕ

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Предмет овог истраживања је студија изводљивости припреме адсорбената од текстилног отпада (полиестера) за уклањање фармацеутских производа као што је клофибринска киселина (КА). Резултати су показали да су адсорбенти припремљени хемијском активацијом у присуству фосфорне киселине, а затим пиролизом на 600 °C, микропорозни материјали са великим специфичним површинама. Испитан је утицај времена контакта, почетне концентрације КА, pH раствора и температуре на адсорпцију. Проценти уклањања КА из воденог раствора адсорпцијом већи од 95% добијени су у случају разблажених раствора (10 mg L^{-1}) на собној температури и при pH=3. Кинетика адсорпције је успешно описана моделом псеудо-другог реда, а добијени изотерме су Фројндлиховог типа. Резултати указују да је овај процес спонтан, ефикасан и потенцијално применљив у уклањању КА из воде.

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